IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

. In re United States Patent Application of:	) Docket No.: 4241-685
Applicants: XU, Xueping, et al.	) Conf. No.: 1430
Application No.: 10/712,351	) Art Unit: 1775
Date Filed: November 13, 2003	) Examiner: Timothy M. Speer
Title: LARGE AREA, UNIFORMLY LOW DISLOCATION DENSITY GAN SUBSTRATE AND PROCESS FOR MAKING THE SAME	) Customer No.: **23448**

DECLARATION OF DR. ROBERT P. VAUDO UNDER 37 C.F.R. § 1.132 IN SUPPORT OF TRAVERSAL OF CLAIM REJECTIONS IN U.S. PATENT APPLICATION NO. 10/712,351

1.   My name is Robert P. Vaudo. I have a PhD. in electrical engineering from North Carolina State University and was a Post-doctoral fellow at Boston University. I have been performing research in wide bandgap semiconductors for approximately 17 years. For more than 12 of those years I have been directly involved in the research and development of wide bandgap III-V nitride substrates, and I am named as an author or co-author of numerous papers published in technical journals relating to such materials. I am currently a Senior Scientist in the Materials Department at Cree, Inc., which is a leading innovator and manufacturer of semiconductors. I am named as a co-inventor on a large number of U.S. patents, foreign patents, and pending patent applications relating to wide bandgap III-V nitride materials, including, for example, U.S. Patent Nos. 7170095, 7118813, 6972051, 6951695, 6943095, 6765240, 6596079, 6533874, 6488767,

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. 6447604, 6440323, and 6156581. I am a co-inventor of the subject matter of the above-referenced patent application.

2.   The claims of the above-referenced patent application (for example, claim 1) recite large area of at least 15 cm^2 and low dislocation density not exceeding 3×10^6 dislocations per cm2 of growth surface area on the face.

3.   I understand that all of the claims of the above-referenced patent application have been rejected. I have reviewed the Final Office Action dated February 17, 2007 and the Office Action dated July 26, 2006 (collectively, "the Office Actions"), along with U.S. Patent No. 6,806,508 to D'Evelyn (hereinafter, "D'Evelyn '508") relied upon by the Examiner in the rejection of the claims. I have further reviewed a paper by M.P. D'Evelyn et al. entitled "Growth and Characterization of Bulk GaN Crystals at High Pressure and High Temperature" (Mat. Res. Soc. Symp. Proc. Vol. 798, Materials Research Society, 2004) (hereinafter, the "D'Evelyn Paper") (attached as **Exhibit A**).   Based on my review of these documents, I have the following comments relating to the Office Actions.

4.   D'Evelyn '508 contemplates "single gallium nitride crystals having a diameter and a thickness in a range from about 0.02 inch (about 0.05 cm) to about 12 inches (about 30 cm) and for example, a size in a range from about 2 inches to about 6 inches" (D'Evelyn '508, col. 4, lines 61-66). This is an extremely wide range, i.e., representing a 600-fold span from the lowest value (e.g., 0.02 inch) to the highest value (e.g., 12 inches).

5.   D'Evelyn '508 purports to teach the fabrication of large area gallium nitride crystals via a "high pressure high temperature" process, with the following excerpt from D'Evelyn '508 representing the most detailed instructions for implementing such grown process:

> More specifically, one suitable process for forming the GaN substrate comprises providing a source gallium nitride, solvent, and mineralizer. The source gallium nitride may comprise at least one of poorly-crystallized gallium nitride, well-crystallized gallium nitride, amorphous gallium nitride, polycrystalline gallium nitride, and combinations thereof. The source gallium nitride may be provided "as-is" in its raw form.   Alternatively, the source gallium nitride can be compacted into a "pill" or sintered into a polycrystalline compact. Alternatively, the source gallium nitride can be formed in situ by providing gallium metal which then reacts with the ammonia solvent after sealing of the capsule and treatment at high pressure and high temperature to form source gallium nitride.
>
> The source gallium nitride may then be combined with at least one of the mineralizer and solvent to form a mixture. The gallium nitride, solvent, and mineralizer may optionally be provided individually to the capsule as separate and distinct un-combined materials. The mixture, which can comprise gallium

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PAGE 22/32 * RCVD AT 4/12/2007 8:45:52 PM [Eastern Daylight Time] * SVR:USPTO-EFXRF-3/17 * DNIS:2738300 * CSID:9194199354 * DURATION (mm-ss):15-30

nitride and at least one of the solvent and mineralizer, can be optionally compacted into a pill, however the compacting of the mixture need not be conducted in the gallium nitride growth process.

The source gallium nitride, solvent, and mineralizer are then placed inside a capsule as either a compacted or uncompacted mixture. Optionally, additional mineralizer can also be added to the capsule. The capsule, which will be described hereinafter, can then be filled with a nitrogen-containing solvent, for example at least one of ammonia or hydrazine, or an organic solvent, including but not limited to, methylamine, melamine, ethylene diamine, and mixtures thereof. The capsule is then sealed, disposed in a pressure cell, and subjected to high pressure and high temperature conditions in an appropriate high pressure high temperature (HPHT) system. The HPHT conditions are maintained for a length of time sufficient to dissolve the source gallium nitride and re-precipitate it onto at least one gallium nitride crystal, gallium nitride boule, or gallium nitride crystal seed.

Maintaining HPHT conditions yields large single gallium nitride crystals, for example single gallium nitride crystals having a diameter and thickness in a range from about 0.02 inch (about 0.05 cm) to about 12 inches (about 30 cm) and, for example, a size in a range from about 2 inches to about 6 inches. The pressure, as embodied by the invention, is in a range from greater than about 5 kbar to about 80 kbar, and the temperature for the gallium nitride crystal growth process is in a range between about 550.degree. C. and about 3000.degree. C. The GaN single crystals thus formed are substantially transparent, with an absorption coefficient below 100 cm.sup.-1. Furthermore, the substrates of the present invention have carrier mobilities above about 100 cm.sup.2 /V-s and strain, with respect to undoped GaN homoepitaxial layers, below about 0.005%.

D'Evelyn '508, col. 4, line 22 – col. 5, line 8 (annotated).

6.   While D'Evelyn '508 purports to teach the fabrication of large area gallium nitride crystals and to further teach the fabrication of low defect density gallium nitride crystals, the disclosure of D'Evelyn '508 gives rise to many critical but unanswered questions regarding fabrication of gallium nitride crystals characterized simultaneously by both large area and low defect density. Such questions include:

> (A)   How is growth nucleated to replicate the seed and prevent inhomogeneity and cracking in the growing GaN crystal according to D'Evelyn's HPHT process?
>
> (B)   How are spurious deposits eliminated so that high quality growth can be carried out for the presumably many days and/or weeks needed to make large area crystals according to D'Evelyn's HPHT process?

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(C)   What measures are taken to eliminate the nucleation of polycrystalline growth or evolving growth conditions with growth of a large crystal according to D'Evelyn's HPHT process?

(D)   How are during growth stresses managed to eliminate cracking, which would reduce the useful size of the resulting crystal grown according to D'Evelyn's HPHT process?

Answers to the foregoing questions (A)-(D) would be necessary for one of ordinary skill in the art to grow GaN crystals characterized simultaneously by large area and low defect density (i.e., within the scope of the claims of the present U.S. Patent Application No. 10/712,351) using the high pressure high temperature process of D'Evelyn '508. Nothing in D'Evelyn '508 provides any answers or guidance relating to any of the foregoing questions (A)-(D).

7.   These gaps in the disclosure of D'Evelyn '508, together with my understanding of the level of ordinary skill in the art at the time U.S. Patent Application No. 09/839,941 (that matured into D'Evelyn '508) was filed, support my conclusion that D'Evelyn '508 fails to enable large area, low defect density GaN within the scope of the claims of the present U.S. Patent Application No. 10/712,351.

8.   Generally speaking, at the time the present invention was made, it was not difficult for one skilled in the art to grow low dislocation density GaN crystals of small size, but it was many magnitudes more difficult to grow GaN crystals characterized by BOTH large area and low dislocation density.

9.   D'Evelyn '508 specifically avoids any recitation of dislocation density achieved by the so-called 'high pressure high temperature' or "HPHT" process as applied to a GaN crystal of any particular size. Likewise, D'Evelyn '508 fails to claim low dislocation density GaN in conjunction with a crystal of any particular size.

10.   D'Evelyn '508 includes claims directed solely to photodetectors, to the exclusion of any claims directed to large area, low dislocation density GaN crystals. One skilled in the art would instantly recognize that the value of patent cover for large area, low dislocation density GaN crystals (which are susceptible to a variety of uses) would be many times greater than the relatively narrow photodetector claims provided in D'Evelyn '508. The failure of D'Evelyn '508 to claim large area, low dislocation density GaN is consistent with D'Evelyn's failure to enable such subject matter.

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11.   In the D'Evelyn Paper, Mr. D'Evelyn (along with the other authors) admits that "[t]he largest [GaN] crystal we have grown to date is 15x18 mm in diameter" using a purported high temperature high pressure process. See D'Evelyn Paper at page 276, bottom paragraph. Moreover, in the final passage, D'Evelyn and his co-authors state their belief "that continued development of the present method will permit low-cost manufacturing of high-quality true bulk GaN boules and wafers larger than 50 mm in diameter." (Emphasis added.) The D'Evelyn Paper was published in 2004 – three years after the filing in 2001 of U.S. patent application 09/839,941 that matured into D'Evelyn '508. If Mr. D'Evelyn (as co-inventor of D'Evelyn '508) had truly enabled GaN crystals of up to 12 inch diameter in 2001 using a high pressure high temperature process, it is bewildering – to say the least – that after three years of additional research, he was unable to produce single crystals of GaN greater than 18 mm (i.e., under 1 inch) diameter in 2004, again using a high pressure high temperature process. Likewise, if Mr. D'Evelyn had enabled 12 inch diameter GaN crystals in 2001, why would "continued development" be necessary in 2004 to support scalability of the high pressure high temperature process to produce GaN wafers larger than 50 mm (2.3 inches)? These facts further support my conclusion that D'Evelyn '508 failed to enable the fabrication of large area, low defect density GaN within the scope of the claims of the present U.S. Patent Application No. 10/712,351.

12.   Moreover, close examination of the D'Evelyn Paper reveals that the authors again took great care to list size and defect (e.g., dislocation) density characteristics of GaN crystals separately so that dislocations and area are not necessarily provided together for any single wafer. A hundred-fold (two order of magnitude) range of defect density, i.e., 10^6 to 10^8 cm^{-2}, was reported for etch pit density in the D'Evelyn Paper (with such a wide range suggesting process inconsistency), but without specific correlation of defect density to any particular wafers. Notably, the largest area wafer (D'Evelyn Paper, page 277, Fig. 4) was "dark" in appearance, which appearance is not consistent with low dislocation density. To the contrary, low defect density GaN should be clear or translucent in appearance. These facts further support my conclusion that Mr. D'Evelyn failed to enable or demonstrate possession of a method for producing large area, low defect density GaN (i.e., within the scope of the claims of the present U.S. Patent Application No. 10/712,351) at the time of filing of U.S. Patent Application No. 09/839,941, which matured into D'Evelyn '508.

13.   Based on the foregoing, there exists no basis for the Examiner's conclusion that D'Evelyn '508 enables the claims of the present patent application

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I declare under penalty of perjury that the facts set forth in this declaration are true and correct, that all statements made of my own knowledge are true, and that all statements made on information and belief are believed to be true. I have been hereby warned that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. § 1001, and that such willful false statements may jeopardize the validity of the application or any resulting registration.

Executed at Durham, North Carolina, this _12ᵗʰ_ day of April 2007.

Robert P. Vaudo

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EXHIBIT A

Growth and Characterization of Bulk GaN Crystals at High Pressure and High Temperature

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D. R. Clarke[b], and R. J. Molnar[c]

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ABSTRACT

We report the growth and characterization of bulk GaN single crystals by temperature-
gradient recrystallization at high pressure and high temperature (HPHT), using apparatus adapted
from that used to synthesize gem-grade diamond crystals. The bulk crystals are grown on seeds
that were synthesized by hydride vapor phase epitaxy (HVPE) and subsequently removed from
their sapphire substrate. Our largest crystals to date are 15×18 mm in diameter; however, the
process is scalable to 50 mm and above. The crystals are transparent and well faceted, and
dislocation densities below 100 cm^{-2} have been achieved. Additional characterization of the
GaN crystals is also presented.

INTRODUCTION

Gallium nitride based optoelectronic and electronic device applications have grown
spectacularly during the past decade, led by light-emitting diodes (LEDs) and laser diodes.
However, the performance and reliability of these devices are compromised by the lack of high
quality, cost-effective native substrates. Threading dislocations, with a typical concentration of
10^9-10^{10} cm^{-2} in heteroepitaxial GaN layers on sapphire or SiC, decrease the efficiency and
maximum intensity of light emission in ultraviolet LEDs [1] and the lifetime of laser diodes [2].

Many laboratories and companies are working on growth of bulk and pseudo-bulk GaN
substrates but no fully satisfactory solution is yet at hand. By far the most mature technology is
the growth of thick quasi-bulk GaN substrates, typically on sapphire, GaAs, or LiAlO$_2$, by
hydride/halide vapor phase epitaxy, or HVPE [3,4]. HVPE is currently capable of routinely
producing free-standing GaN wafers with a dislocation density of about 10^7 cm^{-2}, although
lower values have been reported over at least restricted regions (e.g., within a grain) [4].
However, as a one- or few-at-a-time growth technique, wafers produced by this method are
likely to remain rather expensive. In addition, for a number of device applications, lower
dislocation densities may be required. Growth of GaN in liquid Ga under a high N$_2$ pressure is
capable of growing high-quality platelets, approximately 10 mm × 10 mm × 100 μm in
dimension with dislocation densities in the range of 10-10^5 cm^{-2} [5]. However, the growth
conditions are severe (10-15 kbar pressure), growth rates are rather small, and no one has been
able to demonstrate routine growth of larger crystals by this method, despite extensive efforts by
several groups [5,6]. Interesting results have also been obtained on growth of GaN in Ga-based
alloys under milder pressures [7], but definitive data on the dislocation densities achievable by
the latter methods are not yet available.

After silicon, the electronic crystal grown on the largest scale is α-quartz, at the level of thousands of tons per year. Quartz is grown commercially in supercritical water at about 400 °C and 2 kbar in large autoclaves, and if GaN could be grown by an analogous method the cost could be modest. A number of groups have reported growth of GaN in supercritical ammonia [8], but to date the growth rates and crystal quality achieved have been rather modest.

EXPERIMENTAL

In the present work, we have performed GaN growth in supercritical ammonia, but at considerably higher temperatures and pressures than have been accessible to other groups using conventional autoclaves. Single-crystal GaN seeds were grown by HVPE on sapphire substrates to a thickness of about 200 μm, as described elsewhere [3,9]. The sapphire was then removed by laser-liftoff or by conventional lapping with a diamond powder slurry. One or more GaN seeds, polycrystalline GaN raw material, a mineralizer, and ammonia were then sealed into a capsule. The capsule was incorporated into a high-pressure cell capable of producing a temperature gradient, of the type used in diamond synthesis [10], and placed in a press apparatus, as illustrated schematically in Fig. 1. Crystals were grown at temperatures between 600 and 1000 °C at liquid ammonia fills (% of available volume in the capsule) of about 70-95%. Based on an extrapolation of the equation-of-state data [11], the pressure is estimated as lying between about 5 and 20 kbar, as shown by the solid lines in Fig. 2. Partial dissociation of NH_3 into $\frac{1}{2} N_2 + \frac{3}{2} H_2$ could increase the pressure, as shown by the dashed lines in Fig. 2, calculated using the known equilibrium constant and assuming ideal gas behavior.
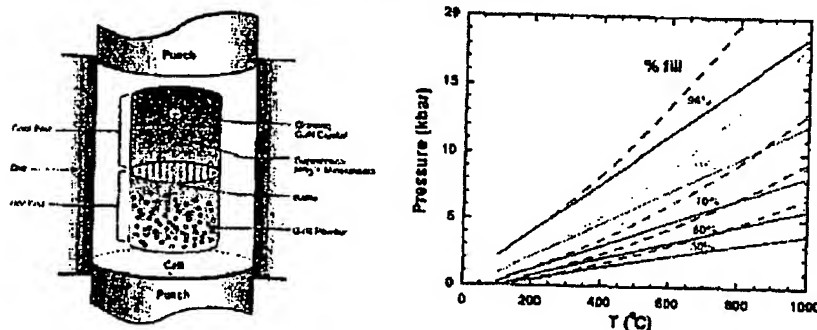
Fig. 1. Schematic of crystal growth apparatus. Fig. 2. Estimated pressure-fill diagram for NH_3.

RESULTS AND DISCUSSION

GaN crystals grown by our method are transparent and well faceted, with roughly similar growth rates in the c- and ab-directions. An example of mm-sized spontaneously-nucleated crystals is shown in Fig. 3. The largest crystal we have grown to date is 15×18 mm in diameter, as shown in Fig. 4. The non-transparency of this crystal results from the seed. A transparent 10×13 mm crystal, grown on a clear seed, is shown in Fig. 5.

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[3,9]. The sapphire was then removed by
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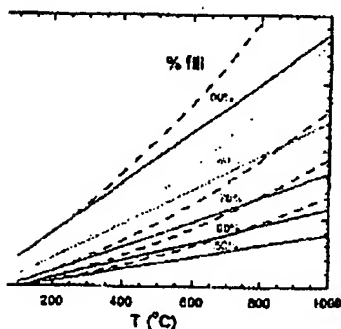
Estimated pressure-fill diagram for NH_3.

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Fig. 3. Spontaneously-
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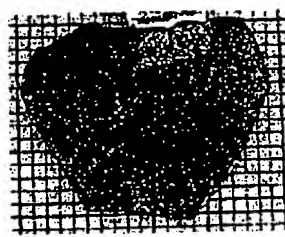
Fig. 4. 15×18 mm crystal
grown on a UCSB seed.
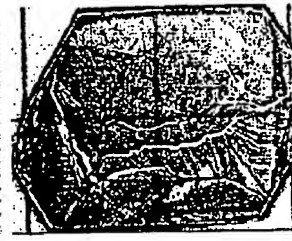
Fig. 5. 10×13 mm crystal
grown on an MIT-LL seed.

The crystals are typically yellow in color rather than colorless, which we attribute to
oxygen, currently present at levels above 100 ppm. The oxygen originates mainly from oxide in
the polycrystalline GaN raw material and from water in the mineralizer, and we expect that it
will be possible to greatly reduce its concentration. An experiment with the purest raw materials
we have produced to date resulted in a GaN crystal that had significantly less of a yellow tint.
Unintentionally doped crystals are n-type but Hall effect data is not yet available.

Under our typical growth conditions GaN crystals typically have one larger and one smaller
polar hexagonal facet, with one set of orthogonally-oriented facets and one set of pyramidal
facets on one end (Fig. 3). We have determined the polarity of the crystals by single-crystal x-
ray diffraction with Mo Kα radiation up to $2\theta = 140°$, or $\sin(\theta)/\lambda = 1.3$Å, comparing the
measured intensities of approximately 2000 reflections, averaged to 280 independent reflections,
to those predicted using the known scattering cross sections of Ga and N. An excellent fit was
achieved, with absolute structure (Flack) parameters of - 0.10(8) and - 0.03(7). The conclusion
of the analysis is that the large hexagonal face is (0001) Ga. Similar and reverse polarities of
prismatic GaN crystals grown in a Na/Ga alloy have been reported [12]. On the macro scale, the
Ga surface is typically very smooth and the N surface somewhat rough.

The surface topography of some as-grown crystals was examined by Atomic Force
Microscopy (AFM; Nanoscope III, tapping mode). A 5×5 μm scan of a Ga face is shown in Fig.
6. The RMS roughness was about 1-3 nm in these scans and was dominated by bumps, which
may be residual particulates or precipitates produced during cooldown from growth conditions.
The N face appeared similar in many regions of the surface, but micron-sized features were
present elsewhere. A crystal was etched in 20% KOH at room temperature and re-examined by
AFM. The Ga face was unchanged while the N face was etched, in agreement with previous
reports [12] and confirming the polarity assignment.

The room-temperature photoluminescence spectrum, excited by a 2 ns nitrogen laser at 337
nm, was dominated by band-edge emission. Raman spectra were taken in back-scattering
geometry with the beam either parallel or perpendicular to the c-axis of the crystal. The results
are shown in Fig. 7. The mode assignments and frequencies of the observed peaks are: A_1 (TO)
530 cm^{-1}; E_1 (TO) 558 cm^{-1}; E_2 (high) 569 cm^{-1}; A_1 (LO) 734 cm^{-1}; and E_1 (LO) 742 cm^{-1}.
The selection rules and frequencies all agree with the literature to within 1-2 cm^{-1} [14].
Particularly noteworthy are the appearance of unshifted LO peaks and the absence of a broad
peak assignable to phonon-plasmon coupling, which indicate no significant strain and a carrier
concentration below about 10^{17} cm^{-3} [15].

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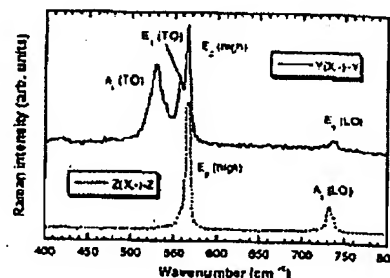Fig. 6. AFM image of as-grown (0001) Ga face. The vertical scale contrast is 0-15 nm.

Fig. 7. Raman spectra of GaN crystal in Y(X,-)-Y and Z(X,-)-Z configurations.

The dislocation densities of the crystals have been measured in two ways. Many or most dislocations in GaN constitute non-radiative recombination centers, and may therefore be quantified by cathodoluminescence (CL) imaging [16], which requires no sample preparation. A CL image of a commercial HVPE-grown GaN crystal is shown in Fig. 8. Approximately 10^7 cm^{-2} dark spots are present, in good agreement with other measurements. By contrast, many HPHT-grown GaN crystals are free of dark spots, as shown in Fig. 9.

A second measure of dislocation density has been obtained by defect-selective etching. We have investigated several techniques, notably vapor-phase HCl etching [17], which offers excellent pit delineation without requiring extremely careful tuning of the etch conditions. Etching of the same HVPE sample as that shown in Fig. 8 yielded an etch pit density of 2×10^7 cm^{-2}, in good agreement with the CL results. Etching measurements on the HVPE seeds used in these experiments yielded etch pit densities of $10^7 - 10^8$ cm^{-2}. Many small, spontaneously nucleated HPHT-grown GaN crystals were free of etch pits. GaN crystals grown on HPVE seeds to a thickness of 0.3-1.5 mm typically had an etch pit density of $10^6 - 10^8$ cm^{-2} in the regions above the original seed, as shown in the bottom portion of Fig. 10. However, GaN material that grew laterally with respect to the c-oriented seed was typically free of etch pits, as shown in the upper portion of Fig. 10. The etch pit density in laterally-grown GaN was less than 500 cm^{-2} in every sample that was examined and was typically below 100 cm^{-2}.
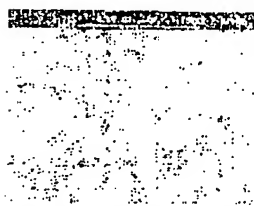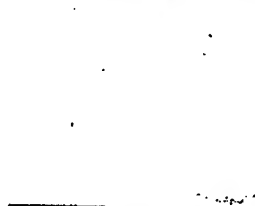

Fig. 8. Cathodoluminescence image of HVPE GaN.

Fig. 9. Cathodoluminescence image of HPHT-grown GaN.

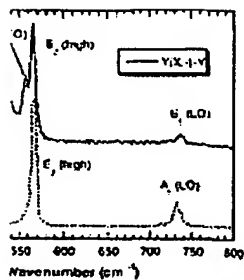Fig. 10. Etched GaN crystal near boundary of underlying HVPE seed.

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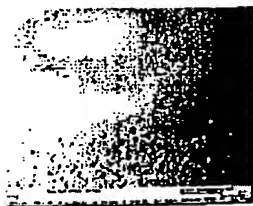N was less than 500 cm^{-2} in

Fig. 10. Etched GaN crystal
near boundary of underlying
HVPE seed.

Spontaneously-nucleated and laterally-grown GaN crystals thus have state-of-the-art dislocation densities and are very high quality in all aspects except for a high impurity content, which is believed to be the source of the observed yellow color. We expect that purification of the polycrystalline GaN and mineralizer raw materials will greatly reduce the impurity levels. The scalability of crystal growth in supercritical fluids is well established, and we believe that continued development of the present method will permit low-cost manufacturing of high-quality true bulk GaN boules and wafers larger than 50 mm in diameter.

ACKNOWLEDGMENTS

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